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(3) - LD - MEASUREMENT OF SELECTED CHEM. IN SOIL FROM THE DEAD CREEK SITE ILLINOIS
EPA SPLIT SAMPLES
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MEASUREMENT OF SELECTED CHEMICALS IN SOIL FROM THE DEAD CREEK SITE ILLINOIS EPA SPLIT SAMPLES

INTRODUCTION

Following media reports and subsequent Illinois EPA concern about hazardous chemicals at the Dead Creek site near Sauget, Illinois, personnel from Monsanto's W. G. Krummrich Plant and the Illinois EPA sampled several areas at the site and split the samples. The Monsanto samples were submittted to Environmental Sciences for characterization. Monsanto's concerns about the site arose from reports of high levels of polychlorinated biphenyls and phosphorus, as well as the reported presence of other chemicals, and the proximity of the site to the Krummrich Plant. These samples were taken to give both Monsanto and the Illinois EPA opportunity to confirm the reported levels found in earlier samplings by the Illinois EPA. In addition to polychlorinated biphenyls and phosphorus, several other "families" of chemicals were measured to try to identify or eliminate possible sources of the chemicals at the site.

SUMMARY

Three sediment samples and one well water sample were taken on October 2, 1980 by Monsanto and IEPA representatives. The Monsanto samples were transferred to our laboratory and analyzed for polychlorinated biphenyls, elemental phosphorus, chlorobenzenes, chlorophenols, phosphate esters, and metals (including arsenic and inorganic phosphorus). No elemental phosphorus was detected in any of the samples, which implies that phosphorus is not responsible for the "smoking earth" reported at the site. In addition, no organic chemicals were detected above the detection limits in the well water sample. However, varying amounts of the organic chemicals and metals were measured in the soil samples. One sample contained higher levels of polychlorinated biphenyls and other organic compounds, while the other two samples contained higher levels of metals. The results clearly indicate non-uniform contamination at the Dead Creek site.

DETAILS

Sampling

The three soil and one water samples were collected by Monsanto W. G. Krummrich plant personnel and IEPA personnel and split at the site. The Monsanto samples were transferred to the Environmental Analysis Group. In our laboratory, the sediment samples were handled according to Standard Operating Procedure (SOP) EAN-80-SOP-6, Homogenizing, Subdividing and Preserving Sediment Samples. Portions of the soil samples were transferred to Applied Sciences for the determination of metals and arsenic.

Analytical Procedures

The three soil samples were analyzed for a variety of chemicals using established procedures or methods developed and validated for the chemicals of interest in soil. The following list tabulates the methods which were used.

Analyte	Method No.	Title
Polychlorinated Biphenyls	ES-80-M-28	Determination of Polychlorinated Biphenyls in Soil and Sediment
Chlorinated Benzenes	ES-80-M-29	Determination of Chlorinated Benzenes in Soil and Sediment
Chlorinated Phenols	ES-80-M-30	Determination of Chlorinated Phenols in Soil and Sediment
Elemental Phosphorus (P_4)	ES-80-M-24	Determination of Elemental Phosphorus (P_4) in Soil and Sediment
Phosphate Esters	ES-80-M-5	Determination of Group I Compounds in Sediments . . .
Metals	Ref. 1, 2	Inductively Coupled Plasma (ICP) . . . Method for Trace Element Analysis of Water and Wastes
Arsenic	Ref. 3	Methods for Chemical Analysis of Water and Wastes-Arsenic

All determinations were carried out in strict accordance with these methods, except that the polychlorinated biphenyls, chlorinated benzenes and phosphate esters were measured in extracts from acidified samples to facilitate determination of chlorinated phenols in the same extracts.

The water sample was extracted in accordance with SOP EAN-80-SOP-19, Extraction of Semivolatile Organic Compounds from Water. The levels of polychlorinated biphenyls and phosphorus were determined using the analytical conditions specified in the respective method for soils listed above.

Results

The analytical results for this study are tabulated in Tables I-VI. Each table contains the results for all of the samples for a specific group of compounds. All results for the soils are in ppm (parts per million or ug/g). The results for the water sample are in ppb (parts per billion, ng/g). In general, the stated detection limits are the lowest level at which a given measurement was validated. Levels which are apparently real, but which are below the validated detection limit are presented in parentheses.

Quality Assurance

The quality assurance results (i.e., recovery and precision evaluations) for these samples have been compiled along with those of similar samples analyzed concurrently. These results are reported in Special Study ES-80-SS-27, Measurement of Selected Chemicals in Soil from the Dead Creek Site - Quality Assurance.

REFERENCES

1. Methods for Chemical Analysis of Waters and Wastes, EPA-600/4-79-020, page: Metals-6, Section 4.1.3.

2. Federal Register, Vol. 44, No. 233, December 3, 1979.

3. Methods for Chemical Analysis of Waters and Wastes, EPA-600/4-79-020, Method 206-Arsenic, pages: 206.2-1 to 206.5-2.

TABLE I. PPM LEVELS OF PCBs AND ELEMENTAL PHOSPHORUS (P_4) IN DEAD CREEK SOIL AND WATER SAMPLES

ANALYTE / ES LOG NO. / DATE SAMPLED / LOCATION	0100301 10/2/80 40 yds south of Queeny Ave. Center of Creek	0100303 10/2/80 268 paces south of 0100301	0100305 10/2/80 270 paces south of 0100303	(Water) 0100307 10/2/80 Well at Theresa's Greenhouse, 101 Walnut, Sauget,IL.	0041701 4/16/80 Soil Blank Mo. Bottoms St.Charles,MO.
PCB's (Cl_2 to Cl_6 Homologs)	13,000	240	45	ND < 1 ppb	ND < 1
P_4	ND < 1	ND < 1	ND < 1	ND < 1 ppb	ND < 1

TABLE II. PPM LEVELS OF CHLOROBENZENES IN DEAD CREEK SOIL SAMPLES

ANALYTE / ES LOG NO. / DATE SAMPLED / LOCATION	0100301 10/2/80 40 yds south of Queeny Ave. Center of Creek	0100303 10/2/80 268 paces south of 0100301	0100305 10/2/80 270 paces south of 0100303	0041701 4/16/80 Soil Blank Mo. Bottoms St. Charles, MO.
MONOCHLOROBENZENE	(0.9)	ND < 1	(0.3)	ND < 1
P-DICHLOROBENZENE	370	(0.3)	(0.4)	ND < 1
O-DICHLOROBENZENE	80	(0.6)	1.0	ND < 1
TRICHLOROBENZENES (3)	85	1.6	(0.7)	ND < 1
TETRACHLOROBENZENES (3)	6.1	2.4	(0.4)	ND < 1
PENTACHLOROBENZENE	ND < 1	ND < 1	ND < 1	ND < 1
HEXACHLOROBENZENE	ND < 1	1.2	ND < 1	ND < 1
NITROCHLOROBENZENES (O-, P-)	120	ND < 1	ND < 1	ND < 1

( ) Values in parentheses are below the validated detection limit. However, they represent levels detected with a S/N >2.5 and can be considered semi-quantitative.

TABLE III. PPM LEVELS OF CHLOROPHENOLS IN DEAD CREEK SOIL SAMPLES

ANALYTE / ES LOG NO. / DATE SAMPLED / LOCATION	0100301 10/2/80 40 yds south of Queeny Ave. Center of Creek	0100303 10/2/80 268 paces south of 0100301	0100305 10/2/80 270 paces south of 0100303	0041701 4/16/80 Soil Blank Mo. Bottoms St.Charles,MO.
O-CHLOROPHENOL	3.7	ND < 1	ND < 1	ND < 1
P-CHLOROPHENOL	6.6	ND < 1	(0.9)	ND < 1
2,4-DICHLOROPHENOL	1.2	ND < 1	ND < 1	ND < 1
PENTACHLOROPHENOL	130	ND < 1	1.8	ND < 1

( ) Values in parentheses are below the validated detection limit. However, they represent levels detected with a S/N >2.5 and can be considered semi-quantitative.

TABLE IV. PPM LEVELS OF PHOSPHATE ESTERS IN DEAD CREEK SOIL SAMPLES

ANALYTE / ES LOG NO. / DATE SAMPLED / LOCATION	0100301 10/2/80 40 yds south of Queeny Ave. Center of Creek	0100303 10/2/80 268 paces south of 0100301	0100305 10/2/80 270 paces south of 0100303	0041701 4/16/80 Soil Blank No. Bottoms St. Charles, MO.
DIBUTYLPHENYL PHOSPHATE	330	ND < 1	(0.8)	ND < 1
BUTYLDIPHENYL PHOSPHATE	ND < 1	ND < 1	(0.8)	ND < 1
TRIPHENYL PHOSPHATE	2600	ND < 1	ND < 1	ND < 1
2-ETHYLHEXYLDIPHENYL PHOSPHATE	ND < 1	ND < 1	2.2	ND < 1
ISODECYLDIPHENYL PHOSPHATE	ND < 1	ND < 1	ND < 1	ND < 1
T-BUTYLPHENYLDIPHENYL PHOSPHATE	28	ND < 1	ND < 1	ND < 1
DI-T-BUTYLPHENYLDIPHENYL PHOSPHATE	ND < 1	ND < 1	ND < 1	ND < 1
NONYLPHENYLDIPHENYL PHOSPHATE	ND < 1	ND < 1	ND < 1	ND < 1
CUMYLPHENYLDIPHENYL PHOSPHATE	3.7	ND < 1	ND < 1	ND < 1

( ) Values in parentheses are below the validated detection limit. However, they represent levels detected with a S/N >2.5 and can be considered semi-quantitative.

TABLE V. PPM LEVELS OF METALS IN LEAD CREEK SOIL SAMPLES

ANALYTE / ES LOG NO. / DATE SAMPLED / LOCATION	0100301 10/2/80 40 yds south of Queeny Ave. Center of Creek	0100303 10/2/80 268 paces south of 0100301	0100305 10/2/80 270 paces south of 0100303	0041701 4/16/80 Soil Blank Mo. Bottoms St.Charles,MO.
SILVER	ND <1	42	29	ND < 1
ALUMINUM	1400	5100	5300	5600
BARIUM	770	1200	1300	130
BERYLLIUM	ND < 1	ND < 1	ND < 1	ND < 1
BORON	28	160	100	27
CALCIUM	8500	9200	6200	4600
CADMIUM	5.1	60	55	3.9
COBALT	15	180	120	33
CHROMIUM	25	110	240	19
COPPER	460	28,000	18,000	19
IRON	4700	53,000	30,000	9900
MAGNESIUM	460	2200	2000	2300
MANGANESE	29	170	110	510
MOLYBDENUM	6.1	92	68	11
SODIUM	400	540	410	320
NICKEL	110	2000	1700	39
LEAD	180	2000	1600	50
PHOSPHORUS	2500	13,000	9400	610
ANTIMONY	13	240	160	29
SILICON	73	150	89	110
TIN	18	260	220	18
STRONTIUM	35	230	110	17
TITANIUM	32	110	80	37
VANADIUM	34	140	130	130
ZINC	280	32,000	18,000	56
AR[illegible] (By AA)	[illegible]			

TABLE VI. SUMMARY OF PHOSPHORUS CONTENT (PPM) OF DEAD CREEK SOIL SAMPLES

ANALYTE / ES LOG NO. / DATE SAMPLED / LOCATION	0100301 10/2/80 40 yds south of Queeny Ave. Center of Creek	0100303 10/2/80 268 paces south of 0100301	0100305 10/2/80 270 paces south of 0100305	0041701 4/16/80 Soil Blank Mo. Bottoms St,Charles,MO.
P - ELEMENTAL, By GC/MS	ND < 1	ND < 1	ND < 1	ND < 1
P-INORGANIC, By ICP	2500	13,000	9400	610
TOTAL PHOSPHATE ESTERS, By GC/MS	3000	ND < 10	4	ND < 10

Submitted by:

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MCA 0023935

MEASUREMENT OF SELECTED CHEMICALS IN SOIL FROM THE DEAD CREEK SITE
W. G. KRUMMRICH PLANT SAMPLINGS

INTRODUCTION

Following media reports and subsequent Illinois EPA concern about the hazardous chemicals at the Dead Creek site near Sauget, Illinois, personnel from Monsanto's W. G. Krummrich Plant sampled several areas at the site. Samples were submitted to Environmental Sciences for characterization. Monsanto's concerns about the site arose from reports of high levels of polychlorinated biphenyls and phosphorus, as well as the reported presence of other chemicals, and the proximity of the site to the Krummrich Plant. These samples were taken to give Monsanto opportunity to confirm the reported levels found in earlier samplings by the Illinois EPA. In addition to polychlorinated biphenyls and phosphorus, several other "families" of chemicals were measured to try to identify or eliminate possible sources of the chemicals at the site.

SUMMARY

Eight sediment samples were taken on August 29 (1), September 15 (2), and September 18 (5), 1980 by Monsanto W. G. Krummrich plant representatives. The samples were transferred to our laboratory for analysis. The samples were analyzed for polychlorinated biphenyls, elemental phosphorus, chlorobenzenes, chlorophenols, phosphate esters, and metals (including arsenic and inorganic phosphorus). No elemental phosphorus was detected in any of the samples, which implies that phosphorus is not responsible for the "smoking earth" reported at the site. Varying amounts of the organic chemicals and metals were measured in the soil samples. The results clearly indicate non-uniform contamination at the Dead Creek site.

DETAILS

Sampling

The eight soil samples were collected by Monsanto W. G. Krummrich plant personnel. The Monsanto samples were transferred to the Environmental Analysis Group. In our laboratory, the sediment samples were handled according to Standard Operating Procedure (SOP) EAN-80-SOP-6, Homogenizing, Subdividing and Preserving Sediment Samples. Portions of the soil samples were transferred to Applied Sciences for the determination of metals and arsenic.

Analytical Procedures

The eight soil samples were analyzed for a variety of chemicals using establisned procedures or methods developed and validated for the chemicals of interest in soil. The following list tabulates the methods which were used.

Analyte	Method No.	Title
Polychlorinated Biphenyls	ES-80-M-28	Determination of Polychlorinated Bipnenyls in Soil and Sediment
Chlorinated Benzenes	ES-80-M-29	Determination of Chlorinated Benzenes in Soil and Sediment
Chlorinated Phenols	ES-80-M-30	Determination of Chlorinated Phenols in Soil and Sediment
Elemental Phosphorus (P_4)	ES-80-M-24	Determination of Elemental Phosphorus (P_4) in Soil and Sediment
Phosphate Esters	ES-80-M-5	Determination of Group I Compounds in Sediments. . .
Metals	Ref. 1, 2	Inductively Coupled Plasma (ICP). . . Method for Trace Element Analysis of Water and Wastes
Arsenic	Ref. 3	Methods for Chemical Analysis of Water and Wastes - Arsenic

All determinations were carried out in strict accordance with these methods, except that the polychlorinated biphenyls, chlorinated benzenes and phosphate esters were measured in extracts from acidified samples to facilitate determination of chlorinated phenols in the same extracts.

Results

The analytical results for this study are tabulated in Tables I-VI. Each table contains the results for all of the samples for a specific group of compounds. All results for the soils are in ppm (parts per million or μg/g). In general, the stated detection limits are the lowest level at which a given measurement was validated. Levels which are apparently real, but which are below the validated detection limit are presented in parentheses.

Quality Assurance

The quality assurance results (i.e., recovery and precision evaluations) for these samples have been compiled along with those of similar samples analyzed concurrently. These results are reported in Special Study ES-80-SS-27, Measurement of Selected Chemicals in Soil from the Dead Creek Site - Quality Assurance.

REFERENCES

1. Methods for Chemical Analysis of Waters and Wastes, EPA-600/4-79-020, page: Metals - 6, Section 4.1.3.

2. Federal Register, Vol. 44, No. 233, December 3, 1979.

3. Methods for Chemical Analysis of Waters and Wastes, EPA-600/4-79-020, Method 206 - Arsenic, pages: 206.2-1 to 206.5-2.

TABLE I. PPM LEVELS OF PCBs AND ELEMENTAL PHOSPHORUS (P_4) IN DEAD CREEK SOIL SAMPLES

ANALYTE / ES LOG NO. / DATE SAMPLED / LOCATION	0091541 8/29/80 100' from Judith Ln.	0091542 9/15/80 North Start	0091543 9/15/80 300' from start	0091907 9/18/80 #9	0091908 9/18/80 #10	0091909 9/18/80 #11	0091911 9/18/80 #14	0041701 4/16/80 #15	Soil Blank Mo. Bottoms St.Charles,MO
PCB's (Cl_2 to Cl_6 Homologs)	29	5000	190	4600	150	730	400	280	ND<1
P_4	ND<1	ND<1	ND<1	ND<1	ND<1	ND<1	ND<1	ND<1	ND<1

TABLE II. PPM LEVELS OF CHLOROBENZENES IN DEAD CREEK SOIL SAMPLES

ANALYTE / ES LOG NO. / DATE SAMPLED / LOCATION	0091541 8/29/80 100' from Judith Ln.	0091542 9/15/80 North Start	0091543 9/15/80 300' from start	0091907 9/18/80 #9	0091908 9/18/80 #10	0091909 9/18/80 #11	0091910 9/18/80 #14	0091911 9/18/80 #15	0041701 4/16/80 Soil Blank Mo. Bottoms St.Charles,MO
MONOCHLOROBENZENE	NA	NA	NA	(0.9)	2.0	(0.2)	ND<1	(0.1)	ND<1
P-DICHLOROBENZENE	NA	NA	NA	34	4.0	3.4	2.5	(0.7)	ND<1
O-DICHLOROBENZENE	NA	NA	NA	14	(0.5)	1.1	2.3	(0.2)	ND<1
TRICHLOROBENZENES (3)	NA	NA	NA	22	2.0	5.3	3.5	1.1	ND<1
TETRACHLOROBENZENES (3)	NA	NA	NA	4.0	(0.5)	2.1	(0.7)	(0.6)	ND<1
PENTACHLOROBENZENE	NA	NA	NA	ND<1	ND<1	ND<1	ND<1	ND<1	ND<1
HEXACHLOROBENZENE	NA	NA	NA	ND<1	ND<1	ND<1	ND<1	ND<1	ND<1
NITROCHLOROBENZENES (O-, P-)	NA	NA	NA	ND<5	ND<1	1.2	ND<1	ND<1	ND<1

NA - Not Analyzed

( ) Values in parentheses are below the validated detection limit. However, they represent levels detected with a S/N >2.5 and can be considered semi-quantitative.

TABLE III. PPM LEVELS OF CHLOROPHENOLS IN DEAD CREEK SOIL SAMPLES

ANALYTE / ES LOG NO. / DATE SAMPLED / LOCATION	0091541 8/29/80 100' from Judith Ln.	0091542 9/15/80 North Start	0091543 9/15/80 300' from start	0091907 9/18/80 #9	0091908 9/18/80 #10	0091909 9/18/80 #11	0091910 9/18/80 #14	0091911 9/18/80 #15	0041701 4/16/80 Soil Blank Mo. Bottoms St.Charles
O-CHLOROPHENOL	NA	NA	NA	17	ND<1	1.7	ND<1	ND<1	ND<1
P-CHLOROPHENOL	NA	NA	NA	20	ND<1	1.7	1.4	ND<1	ND<1
2,4-DICHLOROPHENOL	NA	NA	NA	4.6	ND<1	ND<1	ND<1	ND<1	ND<1
PENTACHLOROPHENOL	NA	NA	NA	32	ND<1	1.1	ND<1	ND<1	ND<1

NA = Not analyzed

( ) Values in parentheses are below the validated detection limit. However, they represent levels detected with a S/N >2.5 and can be considered semi-quantitative.

MCA 0023940

TABLE IV. PPM LEVELS OF PHOSPHATE ESTERS IN DEAD CREEK SOIL SAMPLES

ANALYTE / ES LOG NO. / DATE SAMPLED / LOCATION	0091541 8/29/80 100' from Judith Ln.	0091542 9/15/80 North Start	0091543 9/15/80 300' from start	0091907 9/18/80 #9	0091908 9/18/80 #10	0091909 9/18/80 #11	0091910 9/18/80 #14	0091911 9/18/80 #15	0041701 4/16/80 Soil Blank Mo. Bottoms St.Charles,MO.
DIBUTYLPHENYL PHOSPHATE	ND<1	ND<100	ND<10	60	ND<1	ND<1	1.0	ND<1	ND<1
BUTYLDIPHENYL PHOSPHATE	NA	NA	NA	ND<1	ND<1	ND<1	ND<1	ND<1	ND<1
TRIPHENYL PHOSPHATE	(0.3)	150	18	200	3.0	ND<1	ND<1	ND<1	ND<1
2-ETHYLHEXYLDIPHENYL PHOSPHATE	3.5	17	11	ND<1	ND<1	1.0	(0.5)	ND<1	ND<1
ISODECYLDIPHENYL PHOSPHATE	ND<1	ND<100	ND<10	ND<1	ND<1	ND<1	ND<1	ND<1	ND<1
T-BUTYLPHENYLDIPHENYL PHOSPHATE	ND<1	ND<100	ND<10	ND<1	ND<1	ND<1	ND<1	ND<1	ND<1
DI-T-BUTYLPHENYLPHENYL PHOSPHATE	NA	NA	NA	ND<1	ND<1	ND<1	ND<1	ND<1	ND<1
NONYLPHENYLDIPHENYL PHOSPHATE	ND<2	ND<200	ND<20	ND<1	1.0	ND<1	ND<1	ND<1	ND<1
CUMYLPHENYLDIPHENYL PHOSPHATE	ND<1	ND<100	ND<10	2.6	2.4	2.4	2.2	2.6	ND<1

MCA 0023941

NA - Not analyzed

( ) Values in parentheses are below the validated detection limit. However, they represent levels detected with a S/N >2.5 and can be considered semi-quantitative.

TABLE V. [illegible] LEVELS OF METALS IN BEAR CREEK SOIL SAMPLES

ANALYTE	ES LOG NO. / DATE SAMPLED / LOCATION	0091541 8/29/80 100' from Judith Ln.	0091542 9/15/80 North Start	0091543 9/15/80 300' from start	0091907 9/18/80 #9	0091908 9/18/80 #10	0091909 9/18/80 #11	0091910 9/18/80 #14	0091911 9/18/80 #15	0041701 4/16/80 Soil Blank Mo. Bottoms St.Charles,MO.
SILVER		17	ND<1	3.3	ND<1	20	20	19	4.2	ND<1
ALUMINUM		2300	720	720	2700	2400	3100	3600	3900	5600
BARIUM		210	2000	640	2400	230	940	1000	1100	120
BERYLLIUM		ND<1	ND<1	ND<1	ND<1	ND<1	ND<1	ND<1	ND<1	ND<1
BORON		68	13	21	36	100	78	76	72	27
CALCIUM		2500	2700	2200	13,000	14,000	6200	9200	5600	4600
CADMIUM		60	5.9	17	5.1	40	42	45	53	3.9
COBALT		67	8.2	13	30	120	85	89	81	33
CHROMIUM		44	19	61	29	88	110	130	51	19
COPPER		25,000	2700	16,000	590	8900	13,000	12,000	14,000	19
IRON		24,000	2000	2600	8700	31,000	28,000	28,000	28,000	9900
MAGNESIUM		1000	400	310	1300	1700	1700	2400	2100	2300
MANGANESE		45	15	9.3	60	210	91	140	90	510
MOLYBDENUM		63	9.5	38	11	54	39	38	47	11
SODIUM		350	690	710	420	510	400	440	360	320
NICKEL		950	140	260	120	1100	900	1100	1400	39
LEAD		1000	390	1400	150	1200	1000	1100	1500	50
PHOSPHORUS		4400	770	2400	1900	7400	7000	6500	6700	610
ANTIMONY		130	23	54	22	160	93	88	120	29
SILICON		210	320	270	94	83	91	63	95	110
TIN		76	27	71	19	71	78	91	62	18
STRONTIUM		64	35	42	24	130	120	110	81	17
TITANIUM		49	60	94	36	56	50	47	51	37
VANADIUM		46	13	14	67	120	92	100	110	130
ZINC		20,000	1400	5900	380	19,000	11,000	10,000	18,000	56
ARSENIC (By AA)		NA	NA	NA	180	50	90	50	30	5

TABLE VI. SUMMARY OF PHOSPHORUS CONTENT (PPM) OF DEAD CREEK SOIL SAMPLES

ANALYTE / ES LOG NO. / DATE SAMPLED / LOCATION	0091541 8/29/80 100' from Judith Ln.	0091542 9/15/80 North Start	0091543 9/15/80 300' from start	0091907 9/18/80 #9	0091908 9/18/80 #10	0091909 9/18/80 #11	0091910 9/18/80 #14	0091911 9/18/80 #15	0041701 4/16/80 Soil Blank Mo. Bottoms St.Charles,MO
P - ELEMENTAL, By GC/MS	ND<1	ND<1	ND<1	ND<1	ND<1	ND<1	ND<1	ND<1	ND<1
P-INORGANIC, By ICP	4400	770	2400	1900	7400	7000	6500	6700	610
TOTAL PHOSPHATE ESTERS, By GC/MS	4	170	29	260	6.4	3.4	3.7	2.6	ND<10

Submitted by:

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MEASUREMENT OF SELECTED CHEMICALS IN SOIL FROM THE DEAD CREEK SITE - QUALITY ASSURANCE

INTRODUCTION

Following media reports and subsequent Illinois EPA concern about hazardous chemicals at the Dead Creek site near Sauget, Illinois, personnel from Monsanto's W. G. Krummrich Plant sampled several areas at the site. Samples were submitted to Environmental Sciences for analyses for polychlorinated biphenyls, elemental phosphorus, chlorinated benzenes, chlorinated phenols, phosphate esters, and metals. During the various determinations, replicates and spiked samples were analyzed to evaluate the performance of the method used for these particular samples.

SUMMARY

This report summarizes the quality assurance results obtained for various samples analyzed during the course of this project. The accuracy (recovery from spiked samples) and precision (relative standard deviation of replicate determinations) results are tabulated herein. Although it would be difficult to summarize the overall performance of the methods for all the analytes, in general, the methods performed at the recovery and precision levels established during method validation.

DETAILS

Analytical Methods

The soil samples were analyzed for the various chemicals using established procedures or methods developed and validated for the chemicals of interest in soil. The following list tabulates the methods which were used.

Analyte	Method No.	Title
Polychlorinated Biphenyls	ES-80-M-28	Determination of Polychlorinated Biphenyls in Soil and Sediment
Chlorinated Benzenes	ES-80-M-29	Determination of Chlorinated Benzenes in Soil and Sediment
Chlorinated Phenols	ES-80-M-30	Determination of Chlorinated Phenols in Soil and Sediment
Elemental Phosphorus (P_4)	ES-80-M-24	Determination of Elemental Phosphorus (P_4) in Soil and Sediment
Phosphate Esters	ES-80-M-5	Determination of Group I Compounds in Sediments . . .
Metals	Ref. 1, 2	Inductively Coupled Plasma (ICP) . . . Method for Trace Element Analysis of Water and Wastes
Arsenic	Ref. 3	Methods for Chemical Analysis of Water and Wastes - Arsenic

All determinations were carried out in strict accordance with these methods, except that the polychlorinated biphenyls, chlorinated benzenes and phosphate esters were measured in extracts from acidified samples to facilitate determination of chlorinated phenols in the same extracts.

Results

The results for the determinations of the compounds of interest have been reported in Special Studies ES-80-SS-24, 25, and 26, Measurement of Selected Chemicals in Soil from the Dead Creek Site . . . This Special Study is a compilation of the quality assurance results for all three Special Studies.

Quality Assurance

The recovery and precision results for the determinations are tabulated in Tables I-V. Each table contains the results for all quality assurance samples for a specific group of compounds. Recovery results are reported as percent recovery, calculated as

$$\% \text{ Recovery} = \frac{\text{Concentration (sample + spike)- average concentration (sample)}}{\text{Concentration (spike added)}} \times 100$$

Precision results are reported as percent relative standard deviation (RSD) for replicate determinations.

The tables present the recovery and precision results in concentration ranges (1-10 ppm to 10,000 - 100,000 ppm). The entries are averages of all values for all samples which had either recovery or precision evaluated in that range. All values are for actual samples except the metals recovery results, which are for spiked blank soil. In the recovery column, NE means Not Evaluated, i.e., no samples were spiked in that concentration range, and ND means Not Determinable, i.e., the spiking level was too low (usually <50%) compared to the level actually in the sample. In the precision columns, NE means Not Evaluated, i.e., no replicates were analyzed which contained the analyte in that concentration range.

More detailed compilations of the accuracy and precision results can be found in Reference 4.

REFERENCES

1. Methods for Chemical Analysis of Waters and Wastes, EPA-600/4-79-020, page: Metals - 6, Section 4.1.3.

2. Federal Register, Vol. 44, No. 233, December 3, 1979.

3. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Method 206 - Arsenic, pages: 206.2-1 to 206.5-2.

4. RGK NBP 1914831

RECOVERY AND PRECISION

TABLE 1. PCBs AND ELEMENTAL PHOSPHORUS (P_4) IN DEAD CREEK SOIL SAMPLES

ANALYTE / LEVEL	1-10 ppm		10-100 ppm		100-1,000 ppm		1,000-10,000 ppm		10,000-100,000 ppm	
	% Rec	% RSD	% Rec	%RSD	% Rec	%RSD	% Rec	%RSD	%Rec	% RSD
PCB's (Cl_2 to Cl_6 Homologs)	ND	NE	70%	17%	120%	18%	77%	58%	NE	0%
P_4	56%	NE	NE	NE	NE	NE	NE	NE	NE	NE

RECOVERY AND PRECISION

TABLE II. CHLOROBENZENES IN DEAD CREEK SOIL SAMPLES

ANALYTE \ LEVEL	1-10 ppm		10-100 ppm		100-1,000 ppm	
	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD
MONOCHLOROBENZENE	105%	NE	110%	NE	100%	NE
P-DICHLOROBENZENE	120%	21%	125%	64%	120%	NE
O-DICHLOROBENZENE	125%	16%	120%	NE	120%	8%
TRICHLOROBENZENES (3)	96%	14%	110%	13%	120%	NE
TETRACHLOROBENZENES (3)	110%	9%	120%	NE	130%	NE
PENTACHLOROBENZENE	140%	12%	120%	NE	140%	NE
HEXACHLOROBENZENE	135%	13%	90%	NE	110%	NE
NITROCHLOROBENZENES (O-,P-)	125%	37%	120%	NE	120%	26%

RECOVERY AND PRECISION

TABLE III. CHLOROPHENOLS IN DEAD CREEK SOIL SAMPLES

ANALYTE / LEVEL	1-10 ppm % Rec	1-10 ppm % RSD	10-100 ppm % Rec	10-100 ppm % RSD	100-1,000 ppm % Rec	100-1,000 ppm % RSD
O-CHLOROPHENOL	19%	34%	64%	NE	58%	NE
P-CHLOROPHENOL	36%	26%	16%	NE	30%	NE
2,4-DICHLOROPHENOL	66%	47%	59%	NE	50%	NE
PENTACHLOROPHENOL	140%	46%	40%	NE	36%	20%

RECOVERY AND PRECISION

TABLE IV. PHOSPHATE ESTERS IN DEAD CREEK SOIL SAMPLES

ANALYTE \ LEVEL	1-10 ppm		10-100 ppm		100-1,000 ppm		1,000-10,000	
	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD
DIBUTYLPHENYL PHOSPHATE	75%	NE	130%	NE	120%	12%	NE	NE
BUTYLDIPHENYL PHOSPHATE	120%	42%	115%	NE	NE	NE	NE	NE
TRIPHENYL PHOSPHATE	120%	89%	120%	NE	115%	NE	NE	6%
2-ETHYLHEXYLDIPHENYL PHOSPHATE	90%	47%	110%	NE	115%	NE	NE	NE
ISODECYLDIPHENYL PHOSPHATE	NE	NE	NE	NE	NE	NE	NE	NE
T-BUTYLPHENYLDIPHENYL PHOSPHATE	70%	NE	92%	100%	100%	NE	NE	NE
DI-T-BUTYLPHENYLPHENYL PHOSPHATE	88%	NE	96%	NE	NE	NE	NE	NE
NONYLPHENYLDIPHENYL PHOSPHATE	84%	8%	76%	NE	96%	NE	NE	NE
CUMYLPHENYLDIPHENYL PHOSPHATE	62%	21%	76%	NE	88%	NE	NE	NE

TABLE V. METALS IN DEAD CREEK SOIL SAMPLES

ANALYTE \ LEVEL	1-10 ppm		1-100 ppm		100-1,000 ppm		1000-10,000 ppm		10,000-100,000 ppm	
	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD
SILVER	NE	NE	NE	46%	NE	NE	NE	NE	NE	NE
ALUMINUM	ND	NE	ND	NE	ND	NE	NE	6.5%	NE	NE
BARIUM	ND	NE	ND	NE	89%	37%	NE	0%	NE	NE
BERYLLIUM	98%	NE	98%	NE	94%	NE	NE	NE	NE	NE
BORON	0%	NE	65%	19%	81%	3%	NE	NE	NE	NE
CALCIUM	ND	NE	ND	NE	ND	NE	NE	8.3%	NE	7.5%
CADMIUM	89%	5.2%	97%	21%	96%	NE	NE	NE	NE	NE
COBALT	51%	NE	115%	6.5%	97%	5.1%	NE	NE	NE	NE
CHROMIUM	27%	NE	109%	20%	91%	6.4%	NE	NE	NE	NE
COPPER	0%	NE	143%	66%	90%	NE	NE	NE	NE	11%
IRON	ND	NE	ND	NE	ND	NE	NE	NE	NE	8.1%
MAGNESIUM	ND	NE	ND	NE	ND	NE	NE	7.8%	NE	NE
MANGANESE	ND	NE	ND	13%	ND	10%	NE	NE	NE	NE
MOLYBDENUM	53%	NE	83%	11%	83%	NE	NE	NE	NE	NE
SODIUM	ND	NE	ND	NE	ND	11%	NE	NE	NE	NE
NICKEL	0%	NE	108%	14%	91%	13%	NE	4.5%	NE	NE
LEAD	0%	NE	165%	21%	93%	NE	NE	6.5%	NE	NE
PHOSPHORUS	ND	NE	ND	NE	ND	10%	NE	17%	NE	7.9%
ANTIMONY	0%	NE	27%	2.9%	27%	13%	NE	NE	NE	NE
SILICON	ND	NE	ND	NE	0%	49%	NE	NE	NE	NE
TIN	88%	NE	85%	5.6%	96%	5.4%	NE	NE	NE	NE
STRONTIUM	81%	NE	105%	3.3%	94%	6.5%	NE	NE	NE	NE
TITANIUM	ND	NE	99%	30%	30%	1.3%	NE	NE	NE	NE
VANADIUM	ND	NE	ND	13%	120%	11%	NE	NE	NE	NE
ZINC	ND	NE	139%	34%	87%	NE	NE	8.9%	NE	16%
ARSENIC (By AA)	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE

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